PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Electrodecantation of Dilute Suspensions

We, E. I. Du Pont de Nemours and Company, a Corporation organised and existing under the Laws of the State of Delaware, of Wilmington, Delaware, United States of America, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to a method of concentrating dilute suspensions, more particularly to the process of electrodecantation, and still more particularly to the electrodecantation of aqueous suspensoids of polytetrafluoroethylene.

Electrodecantation is the term given to a process of concentrating aqueous colloidal dispersion by passing an electric current through the dispersion. This process is described in British Patent No. 492,030 and by Paul Stamberger, J. Colloid Sci. No. 1, 93—103 (1946). In this process the dilute suspensions are subjected to the action of a horizontal electric field. The electric field causes the charged suspended particles to migrate toward the electrode of opposite charge. Their forward motion proceeds until interrupted by vertical diaphragms which are impervious to the colloid but pervious to the passage of the current. Within a short time, a concentrated layer of colloid is formed on the diaphragm surfaces. The concentrated layer does not lose the sol characteristics of the original dilute suspension. The concentrate will either rise or fall to the bottom of the cell, depending on its specific gravity. In either case, it can be collected and will contain all or nearly all of the desired product.

In order to prevent the suspension from coagulating in the concentrating cell and also after concentrating, it has been proposed, to add a dispersing agent to the dilute suspensions before subjecting them to an electric current.

While this method of electrodecantation represents a step forward in the technique of concentrating dilute aqueous colloids, experience in concentrating aqueous suspensions of certain new polymeric materials, such as polytetrafluoroethylene, has shown that further improvements are required in order to obtain the maximum concentration of colloid without destroying the original sol characteristic of the dilute suspension.

It is the primary object of this invention to provide a process and apparatus for concentrating dilute aqueous suspensions continuously at the greatest possible concentration with substantially no tendency toward coagulation. It is a further object to provide a process and apparatus which will greatly increase the efficiency of the prior art electrodecantation cells. A still further object is to provide an apparatus and process which requires a minimum of manual attention once the cell is started in operation. A still further object is to concentrate dilute aqueous suspensoids of polytetrafluoroethylene.

The objects of this invention are accomplished by providing a cell equipped with electrodes, having a plurality of compartments between the electrodes. The separate compartments are formed by vertical diaphragms of a material which is pervious to the passage of an electric current through an electrolyte contained therein but impervious to the colloidal particles of the suspensoid to be concentrated. Means are also provided to continuously feed dilute aqueous suspensoid to the cell and means to continuously collect the concentrate in a receiving reservoir and to automatically return to the concentrating cell a portion of the moderately concentrated suspensoid, which collects at the top of the receiving reservoir and is then drawn off at the bottom of the reservoir.

The invention will be described with specific reference to the concentration of dilute aqueous suspension of polytetra-

fluorethylene, although it is to be understood that it is also useful for concentrating other suspensoids containing colloidal particles suspended in a media through which an electric current can be passed. The following represents a typical formula of an aqueous suspensoid of polytetrafluoroethylene which is well adapted for concentrating by means of the present invention.

	By Wt.
Polytetrafluoroethylene - - -	4.0%
Sodium salt of the sulfuric acid ester of a mixture of long chain alcohols which is predominantly lauryl alcohol -	0.2%
Water - - - - - - - - - -	95.8%

In the accompanying drawing the single figure represents a diagrammatic plan view of the apparatus for carrying out the invention. The apparatus comprises an open cell having rubber or similarly lined walls (1), with vertical stainless steel electrodes (2), connected to a source of direct current such as a motor driven generator (3), through a voltage regulator (3A). The electrodes also serve as end plates for the cell. A current reverser (4) is provided in the circuit to reverse the direction of the current periodically. The cell is divided into a plurality of compartments by means of diaphragms (5). The various compartments are connected at the bottom, top and sides of the cell since the diaphragms (5) do not extend to the bottom, top or sides of the cell. The diaphragm compartments are formed by interlacing a continuous sheet of diaphragm material (5) around vertical supporting means (not shown in sectional drawing taken longitudinally through the center of the cell) located at proper intervals on each side of the cell so that the supporting means are in a staggered arrangement with respect to those on the opposite side in order that the diaphragm surfaces are in planes parallel to the electrodes (2). The terminal diaphragms (5A), immediately adjacent the electrodes, extend to the bottom and sides of the cell, but not the top, and prevent the colloid from contacting and collecting on the electrodes. The diaphragms may consist of parchment paper, cellulosic film more commonly known under the Registered Trade Mark "Cellophane" or any other sheet material which is impervious to the colloid to be concentrated and pervious to the passage of electric current when immersed in an electrolyte.

The dilute suspensoid to be concentrated is introduced in the feed reservoir (6), which is provided with an outlet (7), a feed control valve (8) and a rotameter (9) to measure the rate of feed. The feed flows by gravity to the manifold (10), where it is evenly distributed to the various compartments of the cell. The bottom of the cell, which is trough-shaped, is connected to a concentrate reservoir (11). The top of the concentrate reservoir (11) is connected to the feed line (7) at a point (12) immediately above the cell by means of a recycle line (13) provided with a valve 18. The feed line (7) is constricted as shown at (12), where the recycle line (13) is connected to the feed line to provide a hydraulic lift or suction to the moderately concentrated suspensoid being returned from the concentrate reservoir (11) to the cell for further concentration.

Te cell is charged to capacity by opening the feed control valve (8) and closing outlet valve (17). The current is sent through the suspensoid usually at a potential of about 150 volts at 2 amperes and the colloid particles move horizontally toward the electrode of opposite charge. In the case of aqueous suspensoid of polytetrafluoroethylene stabilized with the sodium salt of the sulfuric acid ester of a mixture of long chain alcohols, which is predominantly lauryl alcohol, the colloid particles are negatively charged and move toward the anode. The forward motion of the particles is arrested by the vertical diaphragms (5), where they are concentrated. As the suspensoid becomes concentrated at the diaphragms, it falls to the bottom of the cell since the specific gravity of the colloid is greater than the suspending media. In order to prevent the colloid from coagulating on the diaphragm (5) as a result of too great a concentration, the current is reversed periodically. When the direction of the current is reversed, the concentrate of polytetrafluoroethylene collected at the diaphragms starts moving in the opposite direction and due to its specific gravity, falls to the bottom of the cell where it is collected in the concentrate reservoir (11). The gases formed at the electrodes (2) resulting from the electrolysis of water produce a foam which is drawn off by suction tubes (16). The rate of feed is adjusted so that only clear supernate flows over the terminal diaphragms (5A) and is discharged through outlet (15) equipped with levelling tube (15A). As the clear supernate flows through the electrode chambers formed by the electrodes (2) and terminal diaphragm (5A), it flushes away the undesirable products of electrolysis.

By proper adjustment of the potential

gradient (volt/centimeter) and the frequency of the current reversal, the colloids are concentrated at the bottom of the cell without destroying the original sol characteristics of the original suspensoid. The concentrate is drawn from the bottom of the cell through discharge line (14), which extends to a point just above the bottom of the concentrate reservoir (11). There is a displacement of moderately concentrated suspenoid which collects in reservoir (11) by the more highly concentrated material, the moderately concentrated suspensoid being recycled to the cell through recycle line (13) for further concentration. The recycle line (13) is connected to the feed line (7) at a point immediately above the cell and below the feed reservoir (6). The flow of the dilute suspensoid in feed line (7) through the constricted section of the feed line as shown at (12) creates a hydraulic lift on the material in recycle line (13) and causes the moderately concentrated suspensoid to continuously flow from the top of the concentrate reservoir (11) to the feed line (7).

The concentrated suspensoid, contains from 55% to 74% polytetrafluoroethylene and is continuously drawn off from the concentrate reservoir (11). Approximately 74% concentrate is the maximum obtainable concentration which will retain the original sol characteristics of dilute suspensoid and not coagulate.

There is no limit on the number of diaphragms which may be inserted in a cell other than the limit imposed by structural arrangement. The diaphragms should be as close together as possible, the distance being limited only by structural limitations. The power consumption is independent of the number of diaphragms in the cell but is dependent upon the distance between the electrodes, the cross sectional area of the electrodes, the electrical resistance of the liquid between the electrodes and the difference of potential applied to the electrodes. The lower the potential, the less power consumed. Obviously, the greater the number of diaphragms, the greater the efficiency of the cell.

EXAMPLE I.

A specific example of an operating condition for concentrating polytetrafluoroethylene suspenoid of 2% solids is as follows. A laboratory cell comprising stainless steel electrodes situated 13.3 centimeters apart and a plurality of diaphragms spaced equally apart from each other and having an effective area of 1320 square centimeters was charged with a 2% aqueous suspensoid of polytetrafluoroethylene containing .1% wetting agent (sodium salts of sulfuric acid esters of a mixture of long chain alcohols consisting predominantly of lauryl alcohol) and water 97.9%. The voltage applied to the electrodes was 32 volts, yielding a potential gradient of 2.4 volts per centimeter. The direction of the current was reversed every five minutes. The rate at which a 60% suspensoid was collected from the cell corresponded to 75 millimeters per hour when the rate of feed of the 2% suspensoid was 1.5 liters per hour.

EXAMPLE II.

A semi-works cell comprising stainless steel electrodes 76 centimeters apart and a plurality of diaphragms spaced equally apart from each other and having an effective area of approximately 13840 square centimeters, was charged with a 4% aqueous suspensoid containing .2% wetting agent (sodium salts of sulfuric acid esters of a mixture of long chain alcohols consisting predominantly of lauryl alcohol) and 95.8% water. The voltage applied to the electrodes was 150 volts, yielding a potential gradient of 1.97 volts per centimeter. The direction of the current was reversed every three minutes. The rate at which a 62% suspensoid was collected from the cell corresponded to 402 millimeters per hour when the rate of feed of a 4% suspensoid was 9.88 liters per hour, resulting in a 94.8% yield of dispersed polytetrafluoroethylene in concentrated form.

The advantages of the present invention over the prior art are:—

1. The apparatus requires about $^1/_{20}$ as much attention as the prior art apparatus, which does not provide means for recycling the lower solids portion of the concentrate to the cell.

2. The danger of coagulating the dispersion due to build-up of concentrate in the cell is greatly reduced.

3. Moderately concentrated suspensoid is automatically recycled and concentrated to a higher solids content.

4. The rate of output of the cell is increased approximately 100% over the rate obtained with a cell not equipped with means to recycle a portion of the concentrate.

The invention has been described with respect to concentrating dilute aqueous suspensoid of polytetrafluoroethylene, although it is to be understood that the invention is also useful for concentrating other suspensoids in which the colloid is dispersed in a medium capable of carrying an electric current.

The invention is also useful for separating the dispersed phase of latices from the suspending medium, as in the case of

natural and synthetic rubber latices in those cases where the specific gravity of the suspended material is greater than that of the suspending medium. The principle of the invention can be applied to separating the dispersed phase from the dispersing medium of any emulsion where the specific gravity of the dispersed phase is greater than that of the suspending medium, in which the diaphragms of the cell are impervious to the dispersed phase and pervious to the suspending medium, and in which case the dispersing medium is capable of transmitting an electric current.

It is apparent that many widely different embodiments of this invention may be made without departing from the scope thereof, and therefore, it is not intended to be limited except as indicated in the appended claims.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. An apparatus for the electrodecantation of a dilute colloidal suspension in which the suspended particles have a specific gravity greater than that of the suspending medium, comprising a cell having spaced electrodes therein, means to apply a difference of direct current potential between the said electrodes, means to reverse the direction of current, a plurality of diaphragms between said electrodes spaced apart from each other, the said diaphragms being impervious to the colloids and pervious to passage of electric current, means to feed dilute aqueous suspension to said cell, means to collect concentrated suspensions from said cells, and means to recycle a portion of moderately concentrated suspension to the cell for further concentration.

2. The apparatus of Claim 1 in which the said means to recycle includes an aspirator operated by dilute suspensoid under pressure.

3. The process of concentrating a dilute colloidal suspension in an electrolytic cell having electrodes, means to apply a difference of direct current potential to the said electrodes, means to reverse the direction of current periodically, a plurality of diaphragms spaced apart from each other between said electrodes, said diaphragms being impervious to the colloid but pervious to the suspending medium, whch is capable of transmitting an electric current, which comprises applying a difference of direct current potential between said electrodes, drawing off concentrated colloids from the bottom of said cell in a reservoir, and continuously recycling the moderately concentrated suspensoid which collects at the top of said reservoir to the cell by means of a hydraulic lift created by the flow of the dilute suspension through a constricted area in said feed line.

4. The process of Claim 3 in which the suspension is a dilute aqueous suspensoid of polytetrafluoroethylene.

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